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EFFECT OF NUCLEAR RADIATION ON THE PROPERTIES OF
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The effect of nuclear radiation on the microstructure, elementary-cell dimensions, hardness, and electric conductivity of titanium, molybdenum, and silicon carbides as well as of gadolinium hexaboride is discussed, with photomicrographs, resistivity curves, and tabulated data. Structurally equivalent crystal lattices with strong interatomic bonds are most resistant to neutron irradiation. Microhardness was increased to various degrees, except for silicon carbide which also showed the lowest defect formation but underwent a greater change in resistivity.

Author

The solid refractory compounds (hereafter abbreviated SRC) of the metalloids boron, carbon, nitrogen, and silicon with each other and with the transition and rare-earth metals uranium, thorium, and plutonium are becoming ever more important (Bibl.1, 2, 6 - 8, 14 - 16, 27). These substances, on the one hand, have important physical properties: hardness, wear resistance, high melting point, sufficient strength for most purposes, resistance to liquid and gaseous aggressive agents (Bibl.3, 4), and on the other hand specific nuclear properties: very high or very low cross sections for neutron capture and

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scattering (Bibl.1, 5).

Many physical and chemical properties of SRC have been studied and generalized (Bibl.3, 4, 9 - 13), but the effect of nuclear radiation on these properties remains almost completely uninvestigated. Scanty information on this subject is given in some reviews (Bibl.1, 17 - 21).

The effect of radiation on various physical properties of SRC has been studied for a number of uranium compounds and for several boron-containing SRC (Bibl.22 - 26). In this paper, we present the results of studies on the effects of nuclear irradiation by a reactor (integral flux) on the microstructure, elementary-cell dimension, hardness, and electric conductivity of titanium, molybdenum, and silicon carbides, as well as of gadolinium hexaboride.

1. Preparation of Materials for Experiments

The gadolinium hexaboride was prepared by reduction of gadolinium oxide with boron in vacuo at 1800°C (Bibl.28). The boride so obtained contained 68.3% Gd, 30.3% B, 0.1% Fe, and 0.1% C and, according to X-ray phase analysis, had only a single phase (cubic hexaboride) with a lattice period of $4.1041 \pm 0.0013 \text{ kX}$ [according to the literature it is 4.1078 kX (Bibl.28)].

The titanium carbide was prepared by reduction of titanium dioxide with carbon in vacuo (Bibl.29). The molybdenum carbide was prepared by carburizing metallic molybdenum powder in a tubular graphite furnace (Bibl.9). The titanium carbide contained 80.3% Ti, 19.6% combined carbon, and 0.1% free carbon. The molybdenum carbide contained 93.89% Mo and 6.11% combined carbon. X-ray studies showed them to be, respectively, single-phase carbides TiC and Mo_2C .

Compacts were prepared by sintering the powders by the method of hot-pressing (Bibl.30). To relieve the internal stresses due to the pressing, the

specimens were annealed for 4 hrs at 1800°C in a vacuum of 1.31 kn/m² (knudsen per m²) followed by cooling for 3 hrs. The density of the titanium carbide /61 specimens was 90 - 97% and that of the molybdenum carbide was 88 - 95% (of theoretical). Metallographic analysis showed the specimens to consist of a single phase with polyhedral grain structure.

The specimens were irradiated in the VVR-M reactor of the Institute of Physics, USSR Academy of Sciences. The intensity of the integral flux of thermal neutrons at the point of irradiation was $\sim 10^{13}$ neutrons/cm².sec, and the time for the desired radiation dose was calculated accordingly. The temperature in the irradiation zone was $\sim 50^\circ\text{C}$. The specimens were also exposed to the gamma radiation of the active zone of the reactor.

2. Results of the Experimental Investigation

Effect of radiation on structure. Examination of polished microsections before and after irradiation with an integral dose of 10^{16} n/cm² showed no appreciable effect on the microstructure. Figures 1a, b are photographs of polished microsections of gadolinium hexaboride before and after irradiation with an integral dose of 10^{19} n/cm². To bring out the structure better, the hot-pressed specimens were melted on one side before irradiation, by direct passage of an electric current. As will be seen from the photographs, the irradiation produced an appreciable refining of the grain.

At doses of 10^{18} n/cm² the microstructure of molybdenum carbide also showed appreciable grain refining (Fig.1c, d). The same integral dose, however, had almost no effect on the microstructure of titanium carbide (Fig.1e, f).

To determine the effect of radiation on the dimensions of the unit cells of the specimens, powder patterns were taken with a RKU camera 114 mm in diameter

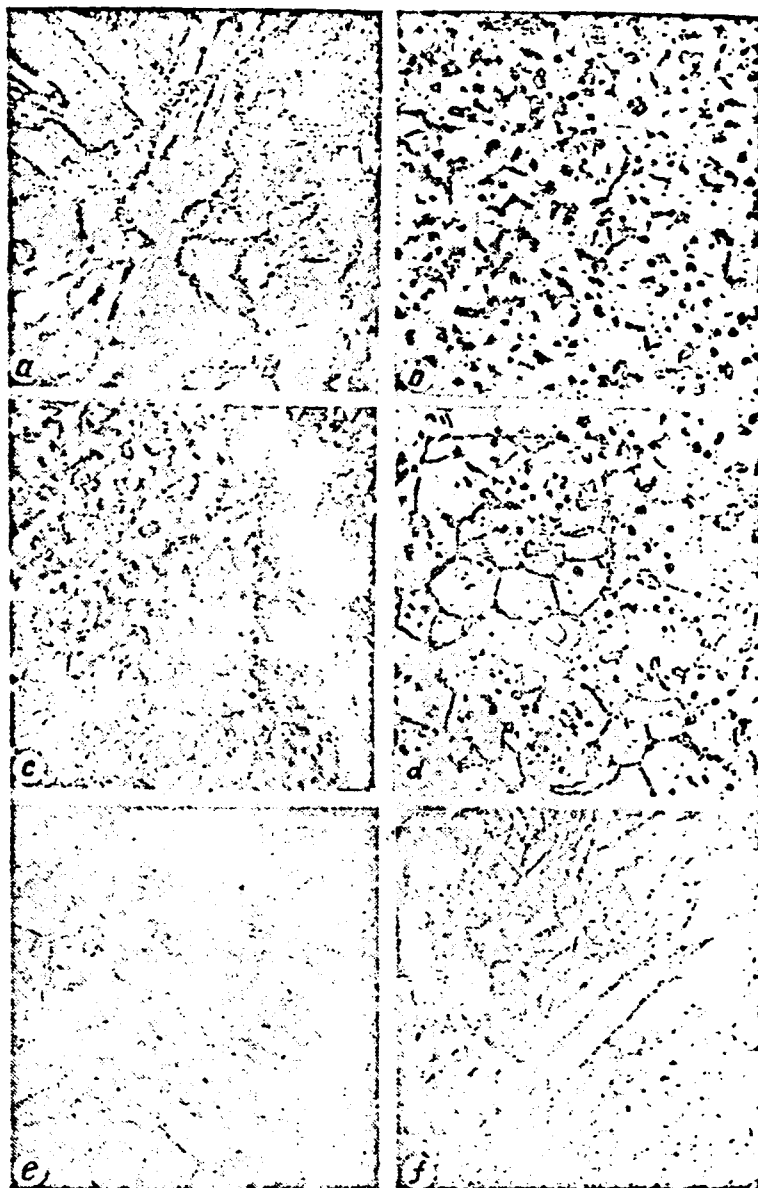


Fig.1 Changes in the Microstructure of SRC Specimens
as a Result of Irradiation

a - GdB_2 before irradiation; b - GdB_2 after irradiation (10^{19} n/cm^2);
c - Mo_2C before irradiation; d - Mo_2C after irradiation (10^{18} n/cm^2);
e - TiC before irradiation; f - TiC after irradiation (10^{18} n/cm^2). $\times 270$

by the diffraction method. Table 1 gives the measurements of the lattice periods before and after irradiation with various doses. Obviously, the lattice period of gadolinium hexaboride increases with the radiation dose, owing to accumulation of radiation defects. The lattice period of the cubic titanium

TABLE 1
LATTICE PERIOD AND RADIATION DOSE

Radiation Dose, n/cm^2	Lattice Period, λ		
	GdB ₆	Mo ₂ C	TiC
Before irradiation	$a=4.1041 \pm 0.0013$	$a=3.016 \pm 0.006$ $c=4.732 \pm 0.009$	$a=4.336 \pm 0.009$
10^{16}	$a=4.1084 \pm 0.0006$	$a=3.016 \pm 0.006$ $c=4.739 \pm 0.009$	$a=4.330 \pm 0.009$
10^{18}	$a=4.1114 \pm 0.0014$	$a=3.016 \pm 0.006$ $c=4.743 \pm 0.009$	$a=4.330 \pm 0.009$

carbide, however, was practically unchanged at the investigated radiation doses. The hexagonal molybdenum carbide shows a distinct increase in the c axis and an unchanged a axis.

The intensity of the X-ray interference lines was measured before and after irradiation of the gadolinium hexaboride specimens, using a URS-50I X-ray ionization diffractometer, and was found to decrease as a result of irradiation, /62 indicating the appearance of additional distortions in the atomic planes.

To define the effects of radiation on the crystal structure of silicon carbide, Laue diffraction patterns of β -SiC single crystals were taken before and after irradiation with an integral flux of $\sim 10^{19} n/cm^2$ (Fig.2). The photographs show that the radiation caused an appreciable asterism of the Laue interferences, due to the distortion of the reflecting atomic planes of the crystal

under the action of neutron bombardment.

/63

Effect of radiation on microhardness. Table 2 gives the most important experimental data on the effect of nuclear irradiation on microhardness. It is

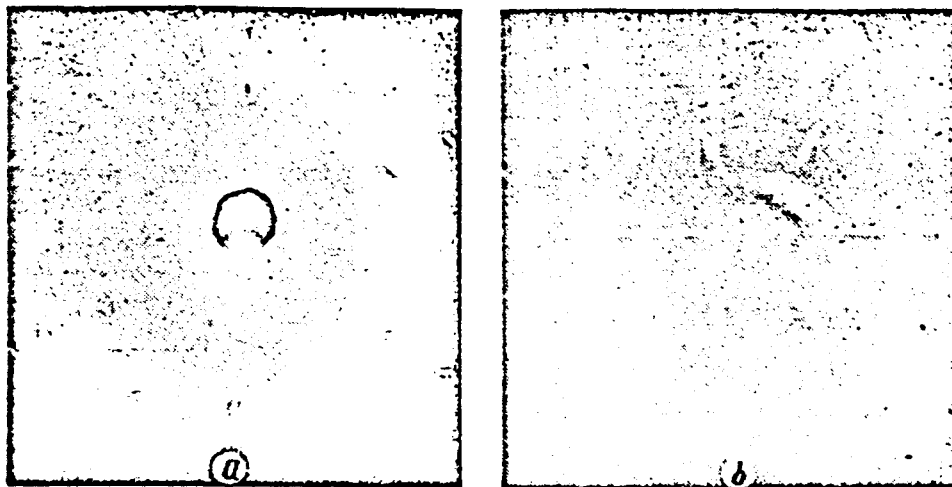


Fig.2 Laue Patterns of Silicon Carbide
a - Before irradiation; b - After irradiation with an
integral dose of $\sim 10^{19}$ n/cm²

apparent that radiation substantially increases the microhardness of gadolinium hexaboride, and that of titanium and molybdenum carbides, but has almost no /64 effect on the hardness of the monocrystalline specimens of silicon carbide.

A study of the effect of annealing on the microhardness of specimens irradiated with a dose of $\sim 10^{18}$ n/cm² showed complete removal of the microhardness due to radiation defects after an anneal at 1000°C for ~ 50 min in the case of Mo₂C, for 65 min in the case of TiC. The microhardness of GdB₂ could be removed only after heating for 4 hrs. Measurements after the anneal show the microhardness to be somewhat below its initial values, as indicated in Table 3.

Effect of radiation on electric conductivity. The electric conductivity of the specimens was measured by a method described elsewhere (Bibl.31), before

and after irradiation, from room temperature to 1000°C. The effect of porosity on the electric resistance was taken into account by the approximation formula proposed in another paper (Bibl.32). Figure 3 gives the resistivity measurements for gadolinium hexaboride before and after irradiation with doses of 10^{16} and 10^{18} n/cm².

TABLE 2
EFFECT OF RADIATION ON MICROHARDNESS

Material	Specimen No.	Microhardness H ₀ before Irradiation	H ₀ after Irrad. by n/cm ²		HX after Irrad. by n/cm ²	
			10 ¹⁶	10 ¹⁸	10 ¹⁶	10 ¹⁸
TiC	6	26.6	35.0	—	31.3	—
	10	25.1	—	34.5	—	37.5
	23	25.6	—	35.4	—	37.3
	26	25.3	32.9	—	31.4	—
Mo ₃ C	4	12.7	—	19.1	—	50.0
	9	12.4	18.2	—	46.5	—
	10	14.6	—	21.3	—	47.7
	12	12.7	18.4	—	44.6	—
Gd ₂ B ₃	2	21.4	30.0	—	40.0	—
	4	20.6	—	32.4	56.8	—
	9	19.5	—	31.8	—	68.3
	15	19.6	—	33.4	70.0	—
SiC	2	56.4	—	58.5	—	—
	3	54.7	—	53.3	—	—

Figure 3 shows that, with increasing radiation dose, both the resistivity and the slope of the temperature versus resistivity curve increase. This indicates, first, that irradiation causes an appreciable additional residual resistance in the Gd₂B₃ specimens, and, second, that the processes of thermal scattering of conduction electrons become more marked with irradiation, due to the

rupture of some interatomic bonds and to a certain relaxation of the lattice.

Table 4 gives the resistivity of titanium and molybdenum carbides for various amounts of radiation. Figure 4 shows the results of a study of the

TABLE 3
MICROHARDNESS OF IRRADIATED CARBIDES AND ANNEALING TIME
AT 1000°C

Carbide	H, before Irradiation 2. Mn/m ² 10 ³	H, dm/mm ² after ir- radiation with a Dose of 10 ¹⁸ n/cm ²	H, Mn/m ² 10 ⁻³ after Anneal for		
			30 min	50 min	65 min
TiC	26.6	35.0	29.7	28.2	26.4
Mo ₂ C № 1	12.4	18.2	14.8	12.3	—
Mo ₂ C № 2	12.7	18.5	14.9	12.4	—

temperature dependence of the resistivity of titanium carbide irradiated by an integral dose of 10^{18} n/cm² and then annealed for 1 hr at 1000°C. As in the

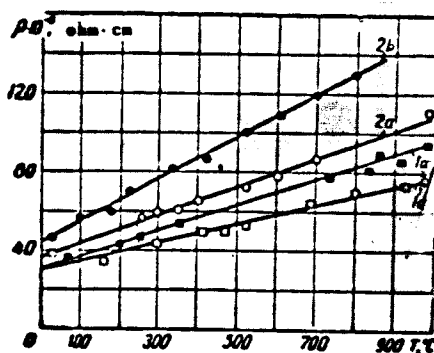


Fig.3 Electric Resistivity of Gadolinium Hexaboride
as a Function of Temperature
1a, 1b - Resistivity of one specimen before and after irradiation
with 10^{16} n/cm². 2a, 2b - Resistivity of another specimen before
and after irradiation with 10^{18} n/cm²

case of gadolinium hexaboride, the slope of the curve of temperature versus resistivity for irradiated specimens of titanium carbide is steeper than

for nonirradiated specimens or for specimens annealed after irradiation, i.e., here too the thermal scattering cross section of the conduction electrons is

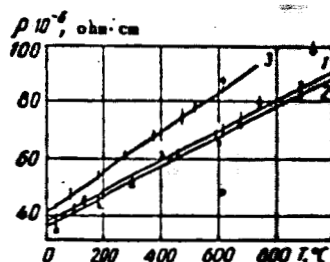


Fig.4 Variation in Electric Resistivity of Titanium Carbide as a Function of Temperature
1 - Before irradiation; 2 - After irradiation with 10^{18} n/cm²;
3 - After irradiation with 10^{18} n/cm² and anneal at 1000°C for 1 hr

increased under the influence of radiation.

The original specimens of silicon carbide single crystals showed a considerable scatter in the values of their resistivity, due to the substantial

TABLE 4
RESISTIVITY OF IRRADIATED CARBIDE SPECIMENS

Radiation Dose n/cm ²	Resistivity μΩ·cm		Relative Increase in Resistivity, %	
	TiC	Mo ₂ C	TiC	Mo ₂ C
Before irradiation	34.0	67.0	—	—
10 ¹⁸	38.7	68.1	13.8	1.7
10 ¹⁹	48.0	75.0	41.2	12.0

influence of relatively small amounts of impurities on the electric properties of a semiconductor. Table 5 shows the changes in the resistivity of silicon /65 carbide specimens on irradiation by an integral neutron flux of 10^{14} - 10^{18} n/cm².*.

* DC measurement by the four-probe method (Bibl.32).

Specimen 1 (Table 5) was a green variety of hexagonal β -SiC. The other SiC specimens were of the black variety. After irradiation by an integral flux of 8×10^{18} n/cm², the green variant of SiC was converted into the black variant.

TABLE 5
RESISTIVITY OF SiC SPECIMENS IN RELATION
TO RADIATION DOSE

Specimen No.	Resistivity (ohm-cm) on Irradiation by an Integral Flux, n/cm ²				
	Before Irrad.	10 ¹⁴	10 ¹⁶	4-10 ¹⁸	8-10 ¹⁸
1	1.85	1.76	—	—	—
2	0.80	0.92	0.57	—	—
3	0.93	0.88	—	—	5.58
4	5.06	5.68	—	—	—
5	3.30	4.20	2.27	—	—
6	1.6	1.4	—	7.5	—

It was impossible to measure its resistivity. Table 5 shows that relatively small doses of radiation (10^{14} - 10^{16} n/cm²) have either no effect or a slightly decreasing effect on the resistivity, whereas larger doses of radiation (10^{18} to 10^{19} n/cm²) result in a substantial increase of resistivity (4 to 6 times), which is considerably more than for the metalloidal carbides (see Table 4).

Effect of radiation on thermal properties. Measuring the linear coefficient of thermal expansion of gadolinium hexaboride showed it to be increased by approximately 25% after irradiation with 8×10^{18} n/cm². Like the increase in the slope of the temperature versus resistivity curve after irradiation, this increase indicates a certain relaxation of the crystal lattice and a /66
weakening of the interatomic bonds under the influence of radiation.

Within the limits of experimental error, however, we found that these radiation doses had no appreciable effect on the linear coefficient of thermal

expansion of the carbide specimens.

3. Discussion of the Results

The conclusion may be drawn from the above experimental data that nuclear radiation has a considerable effect on the electrical, thermal, mechanical and structural properties of the investigated solid refractory compounds (SRC).

A study of these experimental data as to the action of radiation on the crystal structure of SRC indicates that the doses used have the greatest effect on the lattice period of gadolinium hexaboride, which has a rigid three-dimensional framework of covalent bonds between the boron atoms (Bibl.33).

The same doses, however, have only a negligible effect on the lattice period of the cubic titanium carbide and on the lattice period a of the hexagonal molybdenum carbide, where the principal form of the bond is more or less isotropic and of a covalent-metallic character (Bibl.9). However, the period c of the lattice of molybdenum carbide shows a marked increment under irradiation, which increases further with the dose.

The difference in the effect of radiation on the a and c lattice periods is apparently due to the existence of vacancies in the direction of the c axis in the atomic planes of the Mo_2C lattice (Bibl.34), and to the relative ease with which these holes can be filled by molybdenum atoms knocked out by the neutrons, thus increasing the c period. There are no such structurally nonequivalent holes in the direction of the a axis in the atomic planes of Mo_2C , so that the a period is insensitive to relatively small doses of radiation.

The low sensitivity of the lattice period of TiC to relatively small doses of radiation is probably also due to the structural equivalence of all bonds between the equidistant lattice points of TiC , since the lattice is cubic, and

to the greater strength of such bonds. A study, using the URC-50I ionization X-ray diffractometer, showed the intensity of the X-ray interference lines to be somewhat less for the TiC specimens (irradiated with a dose of $\sim 10^{18}$ n/cm²) than for the nonirradiated specimens, indicating that, although the lattice period itself remains unchanged, the radiation still produces a certain distortion in the atomic planes.

From these data, the conclusion should apparently be drawn that structurally equivalent crystal lattices with very strong interatomic bonds are more resistant to the action of nuclear radiation than structurally nonequivalent lattices.

We did not investigate the effect of radiation on the dimensions of the unit cells of silicon carbide, but Laue diffraction patterns of irradiated SiC specimens (cf. Fig.2) show an appreciable distortion of the atomic planes, as also indicated by the results of earlier studies (Bibl.21). Thus, the effect of distortion of the atomic crystalline planes under the action of neutron irradiation is common for all the investigated SRC, except that the relation between their covalent and metallic bonds varies.

The microhardness of all substances investigated by us, except the single crystals of silicon carbide, is considerably increased by neutron bombard- /67
ment. A dose of 10^{19} n/cm² increases the microhardness of GdB₂ by almost 100%. This is evidently due to an accumulation of radiation defects and the attachment of dislocation lines. There is also a tendency for the relative microhardness increment ΔH_r to increase linearly with the radiation dose.

Table 2 shows that, although the nature of the chemical bond in molybdenum and titanium carbides is very similar and is determined mainly by the covalent-metallic interaction Me-C (Bibl.9, 35), exactly the same integral radiation dose

has a greater effect on the hardness of Mo_2C which has a rather anisotropic hexagonal lattice. TiC has a cubic lattice, so that its radiation-induced hardness increment is smaller. On the other hand, the hardness of the cubic GdB_6 increases more under irradiation than the hardness of the cubic TiC or of the hexagonal Mo_2C . Gadolinium hexaboride is characterized by a rigid spatial framework of bonds between the boron atoms, which apparently are of a purely covalent nature (Bibl.36). Thus, the same principle of radiation resistance of solids is confirmed for all three SRC: The degree of change in structure and properties under irradiation increases with the anisotropy of the crystalline structure of the substance and with the approach of the nature of the interatomic bonds to pure covalence.

However, crystals of silicon carbide which has purely covalent interatomic bonds show practically no hardness increment under the radiation doses used. This is obviously due to the fact that, in contrast to TiC , Mo_2C and GdB_6 , the SiC specimens investigated were single crystals, i.e., had a more perfect structure and a lower concentration of dislocations than the polycrystalline specimens of TiC , Mo_2C , and GdB_6 prepared by reduction of the metallic oxides followed by hot-pressing. The radiation-induced point defects of the lattice, to which dislocations are attached, therefore have a considerably greater effect on the radiation-induced hardness increase of these dislocation-saturated polycrystalline specimens than on that of the more perfect specimens of monocrystalline SiC .

It should be remembered that the lattice energy of SiC (17,220 kJ/mole) is considerably higher than that of the other SRC studied (around 8200 kJ/mole) (Bibl.35, 37, 38). Thus, silicon carbide must have the highest threshold energy of all the investigated SRC and the smallest number of radiation-induced atomic

displacements resulting in defect formation (Bibl.40).

Connecting the removal by annealing of the radiation-induced hardness increment with a certain relaxation time τ and assuming that ΔH is due to a change in the elastic energy of the dislocations, the variation of hardness during annealing may be described by the differential equation (Bibl.39):

$$\frac{d}{dt} \left(\frac{\Delta H}{H_0} \right) = \frac{1}{\tau} \left(\frac{\Delta H}{H_0} \right). \quad (1)$$

where H_0 is the equilibrium value of the hardness and t is the annealing time.

Hence,

$$\frac{\Delta H}{H_0} = \left(\frac{\Delta H}{H_0} \right)_{t=0} \exp (-t/\tau). \quad (2)$$

Plotting the relation between hardness and annealing time in semilogarithmic coordinates (Fig.5) indicates that eq.(2) is rather well satisfied for TiC. For Mo_2C , the experimental points are too few to make the same statement more than in first approximation.

The relaxation time τ calculated from Fig.5 is 2×10^3 sec for TiC and 3×10^3 sec for Mo_2C . The adjacent values of the relaxation time for these two carbides correspond to adjacent values of the amplitude of thermal vibrations of the atoms $\sqrt{2} \tilde{U}^{298}$ in these substances, which are 0.085 and 0.090 Å, respectively (Bibl.41). The values obtained for the relaxation times (Bibl.42) seem too great for a simple diffusional displacement of single atoms (of the order of several seconds).

A study of the effect of radiation on the resistivity of the SRC investigated here (Tables 4 and 5) indicates that radiation induces marked changes in the resistivity of both SiC and of metallic SRC - TiC, Mo_2C and Gd_2B_3 . In contrast to the microcut, the change in resistivity of SiC is more pronounced than

in the other SRC which conduct electricity like metals. In this case, a relatively small number of radiation defects (which, judging from the increased resistivity, act as traps for the current carriers) have a substantial effect on the conductivity.

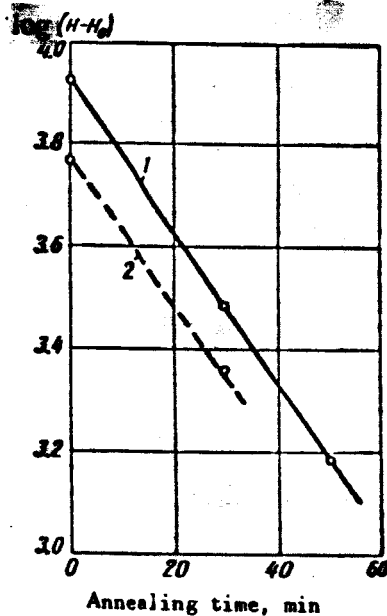


Fig.5 Removal of Radiation-Induced Microhardness of Titanium Carbide (1) and Molybdenum Carbide (2) Versus Annealing Time

The effect of neutron radiation on the resistivity of TiC is greater than its effect on that of Mo₂C, apparently because the neutron-scattering power of the titanium atom is greater than that of the molybdenum atom (Bibl.43), so that the radiation-induced defects in the metallic sublattice of TiC have a greater disturbing potential than in that of Mo₂C. As already noted, the microcut of TiC is, on the contrary, less sensitive to radiation than that of Mo₂C.

The results obtained for the annealing time necessary to eliminate the radiation-induced resistivity and microhardness increments of molybdenum and titanium carbides were somewhat unexpected. In contrast to the results obtained

by other authors (Bibl.44) for the case of metals, the annealing time for these defects was about the same for the various carbides. This question will require further detailed research.

We wish to express our thanks to G.V.Samsonov, corresponding member UkrSSR Academy of Sciences, for his attention to this work and for useful advice, and to E.G.Nikolayev, Candidate in Physical and Mathematical Sciences, Engineer A.A.Galushko, and to V.V.Pen'kovskiy and his associates of the Institute of Physics, UkrSSR Academy of Sciences, for their close cooperation.

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169

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